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# ARC PLASMA DEPOSITION OF NICKEL ZINC FERRITES

RICHARD W. BABBITT

JULY 1972



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# ARC PLASMA DEPOSITION OF NICKEL ZINC FERRITES

by

Richard W. Babbitt

Magnetics, Instrumentation & Interconnections Technical Area
US Army Electronics Technology & Devices Laboratory (ECOM)

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ABSTRACT

Nickel zinc ferrite powders prepared by flame spraying, fluid bed reactions, and a conventional spray dried technique were deposited on ceramic substrates by arc plasma spraying. The deposition rate was dependent upon the powder preparation technique. The magnetic properties evaluated were temperature coefficient, the real part of the permeability tensor, coercive force, and squareness ratio. It was notod that the squareness ratio of arc plasma deposited ferrite was substantially higher than for the same composition conventionally densified. The arc current and the distance from the arc plasma gun to the substrate influenced the values of temperature coefficient, permeability tensor, and coercive force. Measurements of saturation magnetization and resistivity of the deposited ferrite were also made, and the reproducibility of the arc plasma was evaluated. Possible mechanisms for some of the observed phenomena are discussed.

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## INTRODUCTION

The arc plasma, Figure 1, has promise as a new ferrite fabrication technique. But, before its fabrication potential could be explored, it was necessary to develop efficient means of depositing ferrite powders on ceramic substrates and to investigate the effects of the arc plasma operating parameters on the ferrite magnetic properties and their reproducibility. The following information is the experiences and observations of a one year's investigation of the arc plasma technique. The findings, though encouraging, are not meant to be the final answer in the operation or usefulness of the arc plasma, since new effects and procedures are continuing to appear.

Arc plasma spraying is a technique for melting a material and depositing it in a molten state onto a target. The deposited material is rapidly cooled to a solid upon contact with the target. The advantages of the arc plasma over other spraying techniques are its higher operating temperatures (up to 15,000°C) and controlled atmosphere.

Arc plasma deposition being directional in nature, results in a flattening of the molten particles as shown in the electron microscope photograph of Figure 2. When the molten particles hit the target and are cooled, they shrink. This shrinkage can be partially compensated by a yielding of the deposited material or a yielding in the bond to the target; however, a certain amount of stress is expected to be frozen into the deposited particles. When these stresses are severe, cracking of the deposited material or the target occurs. These stresses can be reduced by preheating the target. This minimizes the temperature drop experienced by the molten ferrite as it travels from the gun to the target. The preheating is also helpful in preventing cracking of certain target materials as a result of thermal shock.

It was originally thought that the bond between the deposited coating and the target was mechanical in nature, but more recent information has shown this to be an over-simplification. Currently, there is evidence that the bonding that takes place between the particles of the deposited material is similar to the bonding between the deposited material and the target. For a better bond, it has been found desirable to slightly roughen the target surface before spraying. The roughening process not only cleans the target surface, but tends to restrain and contain the shrinkage of the deposited material.

This report presents data and conclusions on arc plasma depositions of nickel zinc ferrite powders on ceramic substrate targets. The arc plasma deposition of ferrites, recently developed by Harris and Janowiecki,[1,2] presents many problems not previously investigated in the development of the arc plasma technique. Generally, the arc plasma is used for depositing metallized coatings where the reducing effects of the arc plasma are desirable; for the deposition of ferrites, oxidizing effects are desirable. The

stresses resulting from the arc plasma deposition are not only of concern as to their effects on the mechanical properties but also as to their effects on the magnetic properties of ferrites. Thus, before making the arc plasma a useful ferrite fabrication tool, new procedures and the effects of the arc plasma parameters had to be investigated.

This report includes the investigation of deposition techniques, and the effects of arc plasma parameters on magnetic properties, magnetic uniformity, and the reproducibility of the magnetic properties of ferrite materials deposited by the arc plasma technique. The ferrite powders used in this investigation were prepared by conventional techniques with spray dried post treatment, fluid bed reaction, flame spray reaction, and milled sintered bodies. Five ceramic substrates were used as targets: forsterite, magnesium titanate, alumina, $MgO$-$MgAl_2O_4$, and Raytheon's K-38.

The arc plasma parameters which were evaluated include arc current, working distance (distance from arc gun to substrate), and argon versus oxygen as a carrier gas. The magnetic properties evaluated were temperature coefficient (TC), the real part of the permeability tensor ($\mu$), coercive force ($H_c$), saturation magnetization ($4\pi Ms$), and squareness ratio ($Br/_{Bm}$).

## DISCUSSION

### Substrates

#### 1. Cold Substrate

The selection of a proper substrate has an important bearing on the ease with which a ferrite can be deposited. This is especially true when thick films (> 20 mils) are deposited. Ease of deposition implies a wide range of spraying conditions which can be utilized without developing cracks in either the substrate or the deposited ferrite.

The important characteristics to be considered in selecting a ceramic substrate are its susceptibility to damage by thermal shock and the matching of its linear coefficient of thermal expansion to that of the ferrite being deposited. Paladino et al have reported[3] that for hot pressing ferrite and ceramic substrates together, a mismatch in the expansion coefficient of approximately $1x10^{-6}$ per $^{o}C$ causes either cracking of the substrate or the ferrite. Our experience with the arc plasma deposition of ferrite onto ceramic indicates that such a close match in thermal expansion is not necessary, but it is certainly helpful.

The linear coefficients of thermal expansion of several ceramic substrates used in this program are given in Table I. These may be compared to the linear coefficient of expansion of nickel zinc which falls in the range of 9.0 to 10.6 x $10^{-6}$ (for the temperature range of $20^{o}C$ to $320^{o}C$)[4] depending upon the actual composition. These values would be slightly higher for a greater temperature range.

Originally, arc plasma spraying was done with the substrates at room temperature. Three different substrate materials, in two configurations, were used in this phase of the investigation: alumina ($Al_2O_3$), magnesium titanate, and steatite. The two configurations were a flat substrate and a round toroid. The flat substrates were fixed a set distance in front of the arc gun which was manually passed over the substrate surface. The ceramic toroid was mounted on a lathe and spun while the arc gun was moved back and forth over the width of the toroid.

Alumina (by American Lava) was the most widely used substrate in this original phase of the investigation because of the relatively good success achieved with it. Much better success was achieved, with respect to film adherence and thickness, with the toroidal configuration. It was possible to obtain well adhered ferrite films, with thicknesses of up to 30 mils, in about 50% of the toroids. The failures were mostly due to the ferrite film peeling off the toroid, but, in some cases, the ceramic toroids did crack due to thermal shock.

In the case of the flat alumina substrates, it was not possible to deposit a well adhered film greater than 10 mils thick. Normally, as the film thickness approached 10 mils, the film would peel off. Furthermore, in approaching thicknesses of 10 mils on flat substrates, very tight spraying conditions had to be observed (300-500 amperes arc current at a distance of 1½ inch). It was established for both the flat and toroid substrates that, as the arc current was increased and the working distance decreased, there was a tendency for the substrate to crack. When the working distance increased and/or arc current decreased, there was poor film adherence. Roughening the substrate surface by a quick sandblast did aid the adherence somewhat.

Steatite, used as a flat substrate, was similar in behavior to alumina in that with care of arc plasma conditions, it was possible to achieve a well adhered film less than 10 mils thick. Upon polishing the film though, many small surface cracks were observed.

During this original phase, the successful deposition on magnesium titanate (Trans-Tech D-16) was impossible because of the exposure of magnesium titanate to thermal shock. It was not possible, for both toroidal and flat shapes, to get the arc current high enough or the gun close enough for good film adherence without the substrate cracking. Deposition on over twenty flat and toroidally shaped magnesium titanate substrates was attempted without success.

2. <u>Heated Substrate</u>

It became evident that, in order to utilize the wide range of operating parameters available with the arc plasma and to deposit onto ceramic

substrates with a reasonable yield, a modification in the original spraying technique was necessary. The modification was to spray onto a heated substrate. This, it was felt, would overcome most of the substrate cracking due to thermal shock. The technique decided on for heating the substrate was to use an oven with a door which would be opened while spraying, as shown in Figure 3. It was assumed, and later verified, that by proper selection of oven temperature, the heat loss caused by opening the door could be compensated for by the heat of the arc plasma.

The use of the oven reduced substrate cracking; it also enhanced the ferrite to substrate bond. The strength of the bond in most samples was such that any attempts to separate the ferrite from the substrate resulted in either the ferrite or substrate separating from itself, leaving the bonded layer intact. This is not to imply that under some extreme conditions, i.e., working distances greater than 4 inches, arc currents lower than 200 amperes, and poorly prepared substrates, the film would not separate. However, for a wide range of spraying conditions very good bonds were realized. Initially, with the top of the oven opened and the gun fixed in one position, when the substrate was slowly drawn under the arc gun, some cracking occurred in the flat magnesium titanate substrates. It was concluded that when the oven was opened, the part of the substrate which was not immediately passing under the gun cooled sufficiently, such that it was subjected to thermal shock as it passed under the gun. This problem was mostly alleviated by rapidly passing the substrate back and forth under the gun. This procedure maintained the whole substrate near a constant temperature, thus avoiding thermal shock.

An experiment was conducted to determine the temperature of the substrate as it passed under the gun. The substrate was removed and replaced by a thermocouple at the end of a ceramic rod. The thermocouple was moved back and forth in front of the gun at a distance of 1.25 inches and with a stroke of 1 inch. Table II compares these results with the original case where the thermocouple was in a fixed position in front of the gun. It was determined that the temperature was lower in the area transversed by the thermocouple for a given set of spraying conditions. Currently, strokes approaching 2 inches have been found to be satisfactory for substrates of 1 x 2 inches in cross-sectional area.

Using the oven to preheat the substrates, it was possible to deposit films up to 150 mils thick on flat substrates of magnesium titanate, $MgO-MgAl_2O_4$, and forsterite with a yield of greater than 90%. The surprising fact was that alumina, which without the oven was most successful, exhibited several small cracks after cooling and polishing. This was attributed to the thermal expansion mismatch between alumina and the ferrite. Similar characteristics were also noted with steatite. In one extreme case, where a 120 mil ferrite film was deposited on a 100 mil thick steatite substrate, upon cooling, the ferrite bent the steatite enough to crack it. Generally, satisfactory deposition results were realized after the oven was incorporated into the procedure.

## Ferrite Powders

The process used to produce the ferrite powder to be sprayed has a significant influence on the arc current required to produce a dense ferrite film at an acceptable deposition rate. A low arc current is desirable because it allows the substrate and ferrite to be at a lower temperature during deposition. This minimizes the loss of any volatile elements from the powder being deposited. It also eases the problem of matching the thermal coefficients of linear expansion. A rapid deposition rate is important for economic considerations. At the same time, it aids in the deposition of a good film because it decreases the time the substrate is subjected to the heat of the arc plasma gun.

### 1. Particle Size

The size of the ferrite powder particle determines the amount of arc current required for a complete melt of the particle to ensure high density and high deposition rates. Assuming the same powder flow characteristics, smaller particles are more easily carried by a fixed amount of gas flow, but it takes many small particles to achieve the same volume of one large particle. As a consequence, the deposition rate is low; for example, it would take approximately one million 0.1 micron size particles to equal the volume of one 10 micron particle. On the other hand, the larger the particle size, the higher the arc current (more heat) required to achieve a complete melt. The disadvantage of an incomplete melt, where only the outer surface is in a molten state, is that the deposited film will have a low density. Therefore, an optimum particle size for maximum deposition rate must exist, but this has not as yet been established.

Another disadvantage of the very small particles ( 0.2 micron) is that when they are fed into the arc gun, they melt so rapidly that they are in a molten state before leaving the gun nozzle. This creates what is known as loading -- a build up of these particles on the port of the nozzle. When this build up becomes excessive, small pieces break away and are deposited on the target, causing an inhomogeneity in the deposited film. One way to avoid this problem is to use an external powder feed, where the powder is fed in front of the arc gun. The external feed approach with large particles makes a complete melt difficult to achieve. Regardless of the starting particle size, the deposited ferrite has a small X-ray crystallographic size, 0.02 to 0.13 micron.

### 2. Powder Types

The selection of the type of powder to use for an arc plasma production process should include consideration of powder cost, availability, and performance. For this investigation, only performance, which included deposition rate and arc plasma parameters required for good deposition and reproducibility, was evaluated. The nickel zinc ferrite powders used in

this investigation were prepared by flame spray ($Ni_{.52}Zn_{.48}Fe_2O_4$), fluid bed reaction ($Ni_{.50}Zn_{.50}Fe_2O_4$), and spray dried ( $Ni_{.33}Zn_{.67}Fe_2O_4$). The particle sizes of these powders, as determined by an electron microscope, were 0.02 to 0.3 micron, 0.3 to 2.0 microns, and 10 to 100 microns, respectively. The flame spray powders were fully reacted; the fluid bed powders varied from partially reacted to fully reacted; and the spray dried powders were partially reacted. The spray dried powders were processed by Indiana General, Inc., using their process and binder. The apparent advantage in using fully reacted powders is that they are not as susceptable to water absorption.

The deposition characteristics and the ability of the powder to be easily carried by the carrier gas will vary with the amount of moisture the powder contains. The best deposition rates are realized when the ferrite powder is completely dry. It has been observed that when a powder is left in the powder feed hopper during a period of high humidity (80%), its deposition rate is decreased by more than a factor of two. A procedure used to avoid moisture pickup is to store excess powder in a dry place. If moisture is absorbed, the powder should be dried in an oven. It is also felt that a smooth powder particle shape is desirable for rapid deposition rates, since this permits the particles to pass easily over one another.

a. Flame Spray Powder

The flame spray powders which have the smallest particle size were successfully deposited with arc currents from 250 to 500 A at working distances from 1-1/2 to 2-3/4 inches. The deposition rates for these powders were the lowest of the three powder types, 17.0 mils/min/in$^2$ maximum, which was attributed to the small particle size. In one deposition where a 400 A arc current and a 3/4 inch working distance were used, the deposition rate was estimated at 39 mils/min/in$^2$. However, due to the short working distance, the arc flame actually touched the substrate, and the ferrite film was of poor quality. It appeared that the ferrite was in a boiling condition during deposition, and some of the bubbles were quenched into the film after the arc gun was turned off. No further evaluation was made on this film. Densities as high as 99% of theoretical were realized for ferrites deposited from flame spray powders.

b. Fluid Bed Powder

Very few samples were made from the fluid bed powders, since most of the available powders were used in the original phase before the oven was incorporated as part of the system. These few samples were successfully deposited with arc currents from 350 to 600 A at a working distance of 1-½ in. Deposition rates varied from 11 to 23 mils/min/in$^2$ and densities as high as 97% of theoretical were realized.

c. Spray Dried Powder

Several samples were prepared from the commercial spray dried powder. Arc currents from 450 to 720 A with working distances from 1-½ to 2-3/4 in were used. It was observed that when arc currents below 600 A were used, a reddish-brown powder was trapped in the film. This was attributed to an incomplete melt of some of the powder. Deposition rates of up to 60 mils/min/in$^2$ were obtained and densities as high as 94% were measured. Table III shows some of the results obtained by spraying different types of powder on different substrates. Although no specific conclusions can be reached from these data, they are offered for completeness.

3. Arc Current and Working Distance

Deposition rate is also dependent on the selection of arc current and working distance. It has been established that with relatively low arc currents on large working distances, the deposition rate for a given powder decreases as shown in Figure 4. During these tests, it was observed that free powder was coming out of the oven when low arc currents and large working distances were used. It seems that with low arc currents all the particles did not completely melt. Furthermore, several particles resolidified before they hit the target when long working distances were used, thus decreasing the deposition rate. This can also explain why there was a significant improvement in the adhesion of the ferrite to the substrate when the oven was incorporated as part of the deposition technique. The heat of the oven helped prevent the molten particles from cooling as rapidly as when they were being sprayed onto an unheated substrate.

4. Squareness Ratio

The only apparent effect the choice of the powder type had on the magnetic properties was on the squareness ratio (Br/Bm) of the deposited ferrite. This may be somewhat misleading because of the higher zinc concentration in the spray dried powder, and the limited number of fluid bed samples. By using a fixed working distance of 1-½ in, the flame spray and fluid bed powders had higher squareness values than the spray dried powders, as shown in Table IV. Assuming that squareness is increased as a result of stresses produced by the arc plasma process, then it can be explained that the smaller particles of the flame spray and fluid bed powders experience and maintain much more stress upon contact with the substrate because of their complete molten state as compared to the larger spray dried particles. This is verified by the electron microscope results which have shown that only the flame spray and fluid bed particles have a pronounced shape distortion. The squareness for the spray dried powder, though, is still much higher than on conventionally sintered samples as reported in the literature (typically 0.48). This comment is proposed as a general observation and possible explanation for the increased squareness ratio. It was also found that a higher squareness ratio was consistently obtained when the ferrite was annealed at 1280°C for 2 hours

after the substrate had been cut from the ferrite. The results are given in Table V.

## Effects of Arc Plasma Parameters

### 1. Effect of Substrate

The effects of arc current, working distance, and carrier gas on temperature coefficient (TC), $\mu'$, $H_c$, and $4\pi M_s$ were studied. For completeness, it is first desirable to describe the effects of the substrates on these magnetic properties. All samples were toroidal. Some of the samples had the ferrite on toroidal substrates; other samples were ferrite toroids cut free from the ceramic substrate. Some distinct variations in the magnetic properties were noted for these two different sample conditions.

It has already been shown that the ferrites on the substrates exhibited lower squareness ratios than the ferrites cut free from the substrates. It was also found that the ferrites without the substrate exhibited a more positive TC than the ferrite on the substrate even after annealing. Table VI illustrates the effect of the toroidal substrate on the $\mu'$ and $H_c$ characteristics on the deposited ferrite. It may be observed that $\mu'$ of the ferrite on the substrate generally exhibited lower values than the ferrite without the substrate. On the other hand, the coercive force of the ferrites with the substrate generally exhibited higher values than the ferrites without the substrate. It should be noted that annealing did not change the observed trend.

An explanation of this phenomenon is that the mismatch in the linear coefficient of expansion between the ferrite and the substrate affects the ferrite grain growth at the ferrite-ceramic interface during deposition and anneal (stresses are introduced). If this assumption is to have some validity, then the thicker ferrite-substrate samples should have less variation in their magnetic properties than the samples without the substrate. These developed stresses should be primarily limited to the ferrite near the substrate. Figure 5 illustrates the percent variation of $\mu'$ and $H_c$ ($\Delta H_c = H_{c\,without} - H_{c\,with}$; $\Delta\mu' = \mu'_{without} - \mu'_{with}$) after anneal as a function of thickness of the ferrite with the substrate. Due to the limited number of samples tested, a definite conclusion cannot be drawn but a general trend does exist, especially for $H_c$ as a function of thickness.

Since the presence of a substrate has an effect on the magnetic properties, it was necessary to determine the effects of the arc plasma parameters on ferrite toroids with or without substrates. Also, many of the early ferrite deposits were thin (<15 mils), making reliable dimensional measurements difficult. For these samples, TC was used exclusively as the main magnetic property for evaluating the arc plasma parameters, since TC is independent of

dimensional considerations. Furthermore, the measurement of TC is very reliable, with an accuracy of ± 0.1%, and as reported by Dr.Belt, et al, it is also stress sensitive. Therefore, when the thickness of the ferrites was greater than 15 mils, the measurements of $\mu'$ and $H_c$ were considered to be reliable.

The saturation magnetization measurements were only made on deposits thicker than 25 mils, since the ferrite had to be cut free from the substrate for density measurements. Because the small crystallite size in the as deposited ferrites (usually less than 0.1 micron ) would result primarily in a rotational magnetization process, the samples were annealed in order to achieve reasonable values of $\mu'$ and $H_c$.

2. Effects of Arc Current

Each powder type, conventional spray dried, fluid bed, and flame spray, was evaluated separately because of different compositions and particle size.

a. Spray Dried Powder

The samples prepared from the conventional spray dried powder, because of its rapid deposition rate, had thicknesses greater than 15 mils. Therefore, TC measurements were not used exclusively for evaluation. The curves in Figure 6 show the variations in TC with arc current. As expected, the higher arc currents produced a more positive TC. This is similar to the control of TC as developed by a hot pressing technique,[6] where a more positive TC was achieved by higher hot pressing temperatures. Table VII gives the results of how $\mu'$ and $H_c$ responded to a 1200°C anneal for two hours, and how these samples compare to a conventionally sintered (1250°C) sample. It can be assumed that, generally, the higher the arc currents, the higher the $\mu'$ and the lower the $H_c$, before and after anneal. Though the values of $\mu'$ were not as high as the value of the conventionally sintered sample, it is currently felt that a higher $\mu'$ is possible with an improved anneal cycle. The squareness ratios are also included in Table VII to show that no clear trend exists as a function of arc current.

Another interesting observation was that the adjusted $4\pi M_s$ of the deposited ferrite varied from 4100 to 4600 Gauss as compared to 3600 Gauss for the conventionally sintered sample. The adjusted $4\pi M_s$ of the arc plasma samples is the measured $4\pi M_s$ divided by the percent of theoretical density of the sample. This compensates for the density differences which were from 14% to 28% higher than the conventional sample. The conventional sample had a density of 85% of theoretical as compared to 91% to 94% of theoretical density for the arc plasma samples.

b. Fluid Bed Powder

Only a limited number of samples prepared from the fluid-bed

powder were used in the evaluation of the arc plasma parameters because of the limited amount of powder available. The results from these samples were similar to those reported for the conventional spray dried powder. The curves in Figure 7 show the effect of arc current on TC. The fluid bed curves are similar to the conventional spray dried TC curves in that the higher arc currents produce a more positive TC. These values may be compared to a TC of +2000 ppm/°C for a conventionally sintered fluid bed sample. The significance of these TC curves is that it was possible to produce a temperature stable (TC=0) ferrite with a linear characteristic (+0.15%) by arc plasma deposition. This is of economic importance because the production costs of temperature stable toroids by the arc plasma deposition technique are estimated to be from one to two orders of magnitude less than a previously developed flame spray hot press technique.[7] Table VIII shows how $\mu'$ and $H_c$ responded to several arc currents at a working distance of 1-½ in. The values of $\mu'$ and Hc after annealing exhibit an improvement over th conventionally sintered sample which had a low density (60% of theoretical). Also, the toroid, without the substrate, deposited with a 500 A arc current had values of $\mu'$ and $H_c$ similar to those reported in the literature[8] after annealing.

c. Flame Spray Powder

In view of the lower deposition rate of the flame spray powder, ($Ni_{.52}Zn_{.48}$) many of the ferrite deposits were under 15 mils thick and TC was used exclusively to evaluate the arc plasma parameters. Tables IX and X show the effects of arc current and working distance on TC, $\mu'$, and $H_c$. There is a similar trend in the variations of arc current and working distance in that as arc current is increased or working distance is decreased TC becomes more positive, $\mu'$ increases, and $H_c$ decreases. It is felt that the flame spray powder, because of its fine grain size (0.02 to 0.10 micron) and high cost (approximately $25/lb), is the most unsuitable of the three powder types investigated for arc plasma depositions. The fine grain size not only results in a low deposition rate, but makes it difficult to achieve reasonable values of $\mu'$ and $H_c$. The best values of $\mu'$ and $H_c$ obtained were 175 and 2.2 Oe, respectively. These values may be compared to approximately 240 and 1.5 Oe for a similar commercial sample.

3. Carrier Gas

The other arc plasma parameter investigated was the carrier gas. This was done by substituting oxygen for argon; the latter was used in the previous samples discussed. This carrier gas substitution had very little affect on $\mu'$, $H_c$, and squareness ratio, as shown in Table XI. The selection of oxygen as the substitute carrier gas was done specifically to determine the effects on resistivity. It has been established that the presence of ferrous iron in a nickel ferrite will not only decrease the resistivity, but it will also enhance the squareness.[9] This would offer another explanation for the high squareness ratios reported for the arc plasma deposited samples.

The sensitivity of the resistivity of nickel zinc ferrite to ferrous iron, as reported by Smit and Wijn, can be shown by comparing $Ni_{.5}Zn_{.5}Fe_2O_4$ with a resistivity of $10^6$ ohm/cm to $Ni_{.49}Zn_{.49}Fe^{II}_{.02}Fe_2O_4$ which has a resistivity of $10^3$ ohm/cm.
If the arc plasma deposition yielded a high resistivity, approximately $10^6$ ohm/cm, and also a high squareness ratio, then it would have to be assumed that the improved squareness is primarily a result of stress effects. Based on a series of deposited ferrite samples given in Table XII, it is shown that relatively high values of squareness ratios and high resistivities were obtained. Actually, there is no clear relationship between squareness and resistivity. Thus, it is felt that stresses are the main factor in the improved squareness observed in the arc plasma deposited samples. The oxygen carrier gas does appear to have an influence in achieving high resistivities, since the three samples with resistivities greater than $10^6$ ohm/cm were deposited with an oxygen carrier gas.

It is also believed that the loading phenomenon occurring when the fine powders are fed internally into the arc gun has a significant affect on the resistivity of the deposited samples. This is based on observations of consecutive depositions in which those with the greatest loading problem resulted in samples with low resistivities. The sample with the lowest resistivity, $7x10^3$ ohm/cm, which was deposited with an oxygen carrier gas, supports this. Also, two randomly selected samples deposited with an argon carrier gas from the conventional spray dried powder, for which loading was never a problem, had resistivities of $5 \times 10^6$ and $1 \times 10^6$ ohm/cm. More recent work with a Mg Mn ferrite powder, fed externally into the arc flame, produced resistivities greater than $10^9$ ohm/cm. It is concluded that in order to consistently achieve high resistivities, it is necessary to select the proper feed, internal or external, for a given powder particle size and to use an oxidizing carrier gas.

Reproducibility

The reproducibility of arc plasma deposited ferrites was also evaluated. Before reproducibility could be evaluated, it was desirable to first establish the magnetic uniformity of a ferrite deposited on a 1x2 in substrate. To determine magnetic uniformity, two toroids from each deposition were measured and annealed under identical conditions. Table XIII shows the results of six such pairs of toroids. As can be observed, the magnetic uniformity within a single deposition is generally good, both before and after annealing. Therefore, it is felt that a toroid can be cut from any part of the deposited ferrite and possess the magnetic properties representative of the total deposition.

The reproducibility was determined by examining depositions of two or more consecutive samples using identical arc plasma parameters. These depositions fabricated over a time span from as little as a few minutes apart to as long as several days apart. It was observed that when longer

time spans were used between depositions, the deposition rate decreased. It is believed that excessive moisture, picked up during the idle period, contributed to the decrease of the deposition rate. This observation was made after all the samples had been deposited. No attempt had been made to keep the deposition rate constant during the time lapsed depositions. Table XIV shows the results of these experiments which were used to determine reproducibility. The variations of TC, $\mu'$, and $H_c$ are greater than those observed in the determination of magnetic uniformity. This indicates that there is need for improving the control of the arc plasma system. The samples in Table XIV are listed in the chronological order in which they were deposited. It may be stated, therefore, that some improvement in reproducibility has been achieved, during the time span of this investigation. That is, the conventional spray dried samples were deposited early in this investigation while the flame spray samples with TC of +450 and +400 ppm/$^{o}$C were deposited near the end, after several refinements had been introduced in the deposition technique. The values of $\mu'$ and $H_c$ for the samples listed last, seem to indicate better reproducible results. Further improvements in the arc plasma deposition technique will be introduced in the future and it is felt that reproducibility of $\mu'$ and $H_c$ to within $\pm$ 10% is a realistic expectation.

CONCLUSIONS

The most important aspect of depositing ferrite on a ceramic substrate is the preheating of the substrate. The preheat decreases thermal shock, and aids in the ferrite to substrate bond. Further, there is evidence that the linear coefficients of thermal expansion of the substrate and ferrite should be as similar as possible to avoid cracking of either the substrate or ferrite during the time that the composite toroid returns to room temperature. This is believed to be especially important for thick deposits.

The affects the substrate has on the magnetic properties of the deposited ferrite are determined by comparing the properties of ferrite-substrate composite toroids to the properties of ferrite toroids with the substrate removed. It was established that the ferrite-substrate toroids had lower values of $\mu'$, TC, and squareness ratios, and higher values of $H_c$. This effect was most apparent for the thinner ferrite depositions. It would be useful to investigate the variation of the magnetic properties of the ferrite-substrate toroids with linear coefficient of expansion. Also, it would be desirable to know if any differences can be controlled by the cooling rate after the arc plasma deposition and annealing cycle.

The preparation and particle size of the ferrite powder has been found to be the most important factor in achieving high deposition rates. Also, the powder characteristics are important for determining the arc current and working distance necessary to achieve good deposits; i.e., high density, single phase, and good bond. Based on the powder types investigated (flame-spray, fluid-bed, and conventional spray-dried), it is estimated that a

particle size in the range of 1 to 10 microns is best for realizing good deposition rates with moderate arc currents and working distances.

Arc current and working distance were the two arc plasma parameters most thoroughly investigated. They were found to have significant effect on the magnetic properties ($\mu'$, $H_c$, and TC) both before and after annealing. There are limits as how much the working distances can be varied. If the working distance is too short (normally 1-¼ in), the ferrite will remain in a molten state on the substrate during deposition and a tendency exists for the substrate to melt. If the working distance is too long, the ferrite does not adhere to the substrate; this is also the experience with low arc currents. Generally, it is concluded that to achieve a high $\mu'$ and a low $H_c$, a high arc current and short working distance is desirable. The selection of a carrier gas, either inert or oxidizing, had no apparent effect on the magnetic properties but did affect the ferrite resistivity. Another arc plasma parameter which should be investigated is arc gas. Argon was used completely in this investigation. The other possible arc gases are helium, nitrogen, and hydrogen. The use of higher velocity nozzles could also offer significant advantages in lowering the arc currents and increasing the working distances. Greater material stresses would be developed by the higher velocities, resulting in even greater improvement in squareness.

The reproducibility of the arc plasma has been found to be reasonably good, considering the effort expended in this area. It is expected that as further modifications are made the reproducibility will improve.

It is felt that the arc plasma can be a useful technique for the fabrication of specific ferrite components such as temperature stable cores and other components where a ferrite to ceramic bond is required. Also, the arc plasma may be useful for improving certain material characteristics such as squareness and $4\pi Ms$.

ACKNOWLEDGEMENTS

The author expresses his gratitude to J. Klimex for his assistance and dedication in the operation of the arc plasma system, Dr. Rudy Tenzer of Indiana General, Inc. for supplying the commercial spray dried powder, and R. Lent of this Laboratory for several magnetic measurements.

REFERENCES:


1. U.S. Patent 3,576,672, Douglas H. Harris and Richard J. Janowiecki, "Method of Plasma Spraying Ferrite Coatings and Coatings thus Applied," April 27, 1971.

2. D. H. Harris and R. J. Janowiecki, "Arc-Plasma Deposits may Yield Some Big Microwave Dividends," Electronics, February 2, 1970.

3. A. E. Paladino and C. R. Snider, "Ferrite-Dielectric Composites Utilizing $MgO$-$MgAl_2O_4$ Mixtures to Achieve Thermal Expansion Matched Substrates," Ceramic Bulletin, March 1970.

4. F. G. Peters, "Thermal Properties of Polycrystalline Garnets and Ferrites," International Conference on Magnetics, April 1968.

5. Roger F. Belt and Gerald C. Florio, "Permeability and Temperature Coefficient Changes in Hot Press Flame Sprayed Nickel Zinc Ferrites," Presented at 73rd Annual Meeting of The American Ceramic Society, Chicago, Ill., April 26, 1971.

6. Richard W. Babbitt, "Preparation and Reproducibility of Temperature Stable R.F. Ferrites," R&D Technical Report ECOM-3441, June 1971.

7. R. F. Belt and G. C. Florio, "Production Engineering of Ferrite Cores for R. F. Applications," Airtron Division, Litton Precision Products, Morris Plains, N.J., Contract No. DAAB05-69-C-0013, July 1969 - June 1971.

8. J. Smit and H.P.J. Wijn, <u>Ferrites</u> (John Wiley & Sons, New York, N.Y., 1959).

9. U.S. Patent 3,234,136, Junghi Ahn and Pieter Geldermans, "Method for Producing Square Loop Nickel Ferrous Ferrite," February 8, 1966.

Table I

LINEAR COEFFICIENT OF EXPANSION OF CERAMIC SUBSTRATES.

| Substrate | Linear Coefficient of Thermal Expansion (per °C) | | Relative Diel Constant |
|---|---|---|---|
| Alumina (Am. Lava) | 25° - 3000°C<br>25° - 700°C | $6.5 \times 10^{-6}$<br>$7.5 \times 10^{-6}$ | 9.0 |
| Alumina (Trans-Tech, D-9) | | $5 \times 10^{-6}$ | 9.5 |
| Steatite (Am. Lava) | 25° - 300°C<br>25° - 700°C | $8.0 \times 10^{-6}$<br>$8.7 \times 10^{-6}$ | 5.7 |
| Magnesium Titanate (Trans-Tech, D-16) | | $7.5 \times 10^{-6}$ | 16 |
| MgO $MgAl_2O_4$ (Trans-Tech) | | $10 \times 10^{-6}$ | 9.0 |
| Forsterite (Trans-Tech, D-6) | | $10 \times 10^{-6}$ | 6.5 |
| Forsterite (Am. Lava) | 25° - 300°C<br>25° - 700°C | $10.0 \times 10^{-6}$<br>$11.2 \times 10^{-6}$ | 6.0 |
| K-38 (Raytheon) | | $\sim 11 \times 10^{-6}$ | 38 |

Table II

TEMPERATURE PRODUCED BY ARC PLASMA AT A DISTANCE OF 1-1/4 INCHES.

| Arc Current (Amperes) | Thermocouple Temperature (Fixed Position) | Thermocouple Temperature (Transversing w/1 inch Stroke) |
|---|---|---|
| 200 | 840°C | 200°C |
| 400 | 1050°C | 970°C |
| 500 | Thermocouple and alumina casing melted | |

Table III

SUBSTRATES USED FOR ARC PLASMA DEPOSITION.

| Substrate | Arc Current (Amperes) | Work Distance (Inches) | Oven Temp (°C) | | Film Thickness (mils) | Comment |
|---|---|---|---|---|---|---|
| | | | start | during | | |
| **Commercial Spray Dried Powder** | | | | | | |
| Magnesium Titanate | 450 | 1-1/2 | 750 | 720 | 35 | Substrate cracked-Held together by ferrite |
| Magnesium Titanate | 570 | 1-1/2 | 850 | 950 | 25 | Crack-free, well-adhered film |
| Magnesium Titanate | 570 | 1-1/2 | 940 | 1100 | 60 | One crack in substrate |
| Magnesium Titanate | 570 | 2 | 760 | 770 | 61 | One crack in substrate |
| Magnesium Titanate | 570 | 2-3/4 | 950 | 570 | 28 | Crack-free sample |
| $MgO-MgAl_2O_4$ | 620 | 2 | 900 | 880 | 10 | Crack-free, well-adhered film |
| $MgO-MgAl_2O_4$ | 700 | 2 | 890 | 930 | 30 | Crack-free, well-adhered film |
| $MgO-MgAl_2O_4$ | 700 | 2 | 960 | 930 | 36 | Crack-free, well-adhered film |
| **Fluid Bed Powder** | | | | | | |
| Steatite | 350 | 1-1/2 | 730 | 800 | 25 | Cracks in deposited ferrite |
| Magnesium Titanate | 350 | 1-1/2 | 800 | 750 | 10 | Two cracks in substrate |
| Magnesium Titanate | 400 | 1-1/2 | 750 | 830 | 12 | Crack-free, well-adhered film |
| Steatite | 400 | 1 | 750 | 950 | 40 | Cracks in deposited ferrite |
| Magnesium Titanate | 500 | 1-1/2 | 900 | 780 | 22 | Crack-free, well-adhered film |
| Magnesium Titanate | 600 | 1-1/2 | 920 | 1040 | 10 | Crack-free, well-adhered film |

Table III

SUBSTRATES USED FOR ARC PLASMA DEPOSITION (Continu )

| Substrate | Arc Current (Amperes) | Work Distance (Inches) | Oven Temp (°C) start | Oven Temp (°C) during | Film Thickness (mils) | Comment |
|---|---|---|---|---|---|---|
| **Flame Spray Powder** | | | | | | |
| Magnesium Titanate | 300 | 2 | 700 | 580 | 12 | Crack-free sample |
| Magnesium Titanate | 300 | 1-3/4 | 620 | 550 | 4 | Film peeled off-substrate cracked |
| Magnesium Titanate | 300 | 1-3/4 | 600 | 530 | 10 | One crack in substrate |
| Magnesium Titanate | 400 | 1-3/4 | 760 | 680 | 11 | One crack in substrate |
| Magnesium Titanate | 500 | 1-3/4 | 700 | 800 | 34 | Crack-free, well-adhered film |
| Fosterite | 400 | 2 | 870 | 560 | 41 | Poor film adherence-substrate cracked |
| Fosterite | 400 | 1-3/4 | 920 | 700 | 27 | Crack-free, well-adhered film |
| Fosterite | 400 | 1-3/4 | 940 | -- | 32 | Crack-free, well-adhered film |
| Fosterite | 400 | 1-3/4 | 850 | -- | 74 | Crack-free, well-adhered film |

Table IV

SQUARENESS AS A FUNCTION OF POWDER TYPE.

(Working Distance = 1-1/2 In)

| Powder Type | Arc Current (Amperes) | Squareness Ratio |
|---|---|---|
| Flame Spray | 350 | .83 |
| | 400 | .87 |
| | 400 | .89 |
| | 500 | 76 |
| | 500 | .34 |
| Fluid Bed | 350 | .90 |
| | 500 | .87 |
| | 600 | .91 |
| Spray Dried | 450 | .65 |
| | 570 | .84 |
| | 570 | .73 |
| | 600 | .76 |
| | 660 | .73 |
| | 720 | .69 |

Table V

SQUARENESS RATIO AFTER ANNEAL WITH AND WITHOUT SUBSTRATE.

| Arc Current (Amperes) | Working Distance (Inches) | Squareness Ratio | |
|---|---|---|---|
| | | With | Without |
| 400 | 2-1/8 | .77 | .84 |
| 400 | 2-1/8 | .81 | .85 |
| 400 | 1-3/4 | .75 | .82 |
| 500 | 1-1/2 | .76 | .84 |

Table VI

EFFECTS OF SUBSTRATE ON TC, $\mu'$, AND $H_c$.

| Powder Type | Substrate (with or without) | Before Anneal TC (ppm/°C) | Before Anneal $\mu'$ | Before Anneal $H_c$ (Oe) | After Anneal Anneal Temp (°C) | After Anneal $\mu'$ | After Anneal $H_c$ (Oe) | Film Thickness (mils) |
|---|---|---|---|---|---|---|---|---|
| Flame Spray | with | 0 | 16 | -- | 1260 | 78 | 4.2 | 15 |
| | without | +150 | 25 | 27 | 1260 | 175 | 2.2 | 20 |
| Fluid Bed | with | +250 | 38 | 20 | 1240 | 155 | 1.8 | 20 |
| | without | +570 | 46 | -- | 1240 | 203 | 1.2 | 20 |
| Flame Spray | with | +145 | 30 | 18 | 1280 | 130 | 2.8 | 20 |
| | without | +230 | 30 | 18 | 1280 | 138 | 2.0 | 40 |
| Flame Spray | with | +350 | 40 | 13 | 1250 | 88 | 3.8 | 30 |
| | without | +500 | 53 | 13 | 1250 | 128 | 4.0 | 35 |
| Flame Spray | with | -- | 37 | 18.5 | 1310 | 109 | 3.0 | 35 |
| | without | -- | 37 | 18 | 1310 | 118 | 3.0 | 30 |

Table VII

EFFECTS OF ARC CURRENT ON $\mu'$ AND $H_c$ AT A WORKING DISTANCE OF 1-1/2 IN (CONVENTIONAL SPRAY DRIED POWDER).

| Arc Current (Amperes) | Before Anneal $\mu'$ | Before Anneal $H_c$ (Oe) | Anneal Temp (°C) | After Anneal $\mu'$ | After Anneal $H_c$ (Oe) | After Anneal Squareness Ratio | With or Without Substrate |
|---|---|---|---|---|---|---|---|
| 450 | 13.0 | 14.0 | 1200 | 175 | 1.0 | .64 | with |
| 570 | 53.0 | -- | 1200 | 180 | 2.6 | .83 | with |
| 570 | 130.0 | 3.0 | 1200 | 300 | 1.5 | .73 | with |
| 600 | 79.0 | 4.8 | 1200 | 250 | 1.7 | .77 | with |
| 660 | 230.0 | 1.6 | 1200 | 320 | 0.8 | .64 | with |
| 720 | 204.0 | 1.4 | 1200 | 345 | 0.6 | .67 | without |
| Conventional Sintered | | | 1250 | 575 | 0.6 | .50 | without |

Table VIII

EFFECTS OF ARC CURRENT ON $\mu'$ AND $H_c$ AT A WORKING DISTANCE OF 1-1/2 IN (FLUID BED POWDER).

| Arc Current (Amperes) | Before Anneal $\mu'$ | Before Anneal $H_c$ (Oe) | Anneal Temp. (°C) | After Anneal $\mu'$ | After Anneal $H_c$ (Oe) | After Anneal Squareness Ratio | With or Without Substrate |
|---|---|---|---|---|---|---|---|
| 350 | 34 | -- | 1240 | 155 | 2.4 | .90 | with |
| 500 | 38 | 20 | 1240 | 155 | 1.8 | .86 | with |
| 600 | 39 | 19 | 1240 | 135 | 1.8 | .90 | with |
| 500 | 46 | -- | 1240 | 203 | 1.2 | .88 | without |
| Conventional Sintered @1250 | | | | 73 | 2.0 | .73 | without |
| Smit-Wijn[8] | | | | 240 | 1.5 | -- | without |

Table IX

EFFECTS OF ARC CURRENT ON $\mu'$ AND $H_c$ AT A WORKING DISTANCE OF 1-3/4 IN (FLAME SPRAY POWDER (WITH SUBSTRATE)).

| Arc Current (amperes) | Before Anneal | | | Anneal Temp (°C) | After Anneal | | | Film Thickness (mils) |
|---|---|---|---|---|---|---|---|---|
| | TC (ppm/°C) | $\mu'$ | $H_c$ (Oe) | | $\mu'$ | $H_c$ (Oe) | Squareness Ratio | |
| 300 | + 85 | -- | -- | -- | -- | -- | -- | 1.5 |
| 300 | +120 | -- | -- | -- | -- | -- | -- | 2 |
| 350 | 0 | -- | -- | -- | -- | -- | -- | 6 |
| 400 | + 75 | -- | -- | -- | -- | -- | -- | 6 |
| 400 | +150 | -- | -- | -- | -- | -- | -- | 3 |
| 400 | +210 | 28 | 17 | 1280 | 113 | 3.5 | .7 | 30 |
| 500 | +170 | -- | -- | -- | -- | -- | -- | 3 |
| 500 | +400 | 38 | 15 | 1280 | 120 | 3.3 | .8 | 30 |
| 500 | +450 | 45 | 13 | 1260 | 110 | 2.5 | .84 | 20 |

Table X

EFFECTS OF WORKING DISTANCE ON $\mu'$ AND $H_c$ WITH AN ARC CURRENT OF 400 A (FLAME SPRAY POWDER (WITHOUT SUBSTRATE)).

| Working Distance (inches) | Before Anneal | | | Anneal Temp (°C) | After Anneal | | | Film Thickness (mils) |
|---|---|---|---|---|---|---|---|---|
| | TC (ppm/°C) | $\mu'$ | $H_c$ (Oe) | | $\mu'$ | $H_c$ (Oe) | Squareness Ratio | |
| 2-3/4 | -70 | 11 | -- | 1300 | 103 | 5.0 | .78 | 30 |
| 2-3/4 | -110 | 13 | -- | 1300 | 100 | 5.0 | .79 | 18 |
| 2-1/2 | +50 | 14 | -- | -- | -- | -- | -- | 10 |
| 2 | 0 | 16 | -- | 1280 | 113 | 5.0 | .80 | 40 |
| 1-3/4 | +90 | 27 | 21 | 1310 | 144 | 3.1 | .86 | 80 |

Table XI

EFFECTS OF CARRIER GAS ON $\mu'$, $H_c$, AND SQUARENESS RATIO.

ARGON

| Arc Current (amperes) | Working Distance (inches) | Before Anneal | | | After Anneal | | |
|---|---|---|---|---|---|---|---|
| | | $\mu'$ | $H_c$ (Oe) | Anneal Temp (°C) | $\mu'$ | $H_c$ (Oe) | Squareness Ratio |
| 300 | 1-3/4 | 10 | -- | -- | -- | -- | --- |
| 400 | 1-3/4 | 32 | -- | 1330 | 115 | 2.4 | .89 |
| 400 | 1-3/4 | 28 | -- | 1280 | 111 | 3.4 | .70 |
| 400 | 1-3/4 | 25 | 21 | 1310 | 138 | 3.1 | .85 |
| 400 | 1-3/4 | 25 | 22 | 1310 | 120 | 3.5 | .88 |
| **OXYGEN** | | | | | | | |
| 250 | 1-3/4 | 9.5 | -- | 1300 | 97 | 5.2 | .85 |
| 400 | 1-3/4 | 21 | -- | 1300 | 96 | 4.2 | .88 |
| 400 | 1-1/2 | 24 | -- | 1360 | 118 | 2.5 | .89 |
| 400 | 1-1/2 | 24 | 24 | 1360 | 102 | 2.5 | .65 |
| 400 | 1-1/2 | 16 | -- | 1360 | 122 | 3.8 | .80 |

Table XII

COMPARISON OF RESISTIVITY AND SQUARENESS RATIO.

| Resistivity (ohm/cm) | Squareness Ratio | Carrier Gas | Anneal Conditions |
|---|---|---|---|
| $6 \times 10^7$ | .85 | $O_2$ | air anneal - fast cool |
| $4 \times 10^6$ | .72 | $O_2$ | air anneal - fast cool |
| $2 \times 10^6$ | .85 | $O_2$ | air anneal - fast cool |
| $3 \times 10^5$ | .89 | Argon | $O_2$ anneal - slow cool |
| $2 \times 10^5$ | .88 | Argon | air anneal - slow cool |
| $9 \times 10^4$ | .89 | Argon | $O_2$ anneal - fast cool |
| $5 \times 10^4$ | .86 | Argon | $O_2$ anneal - fast cool |
| $5 \times 10^4$ | .88 | $O_2$ | air anneal - fast cool |
| $2 \times 10^4$ | .84 | Argon | air anneal - fast cool |
| $1 \times 10^4$ | .80 | Argon | air anneal - fast cool |
| $7 \times 10^3$ | .90 | $O_2$ | air anneal - fast cool |

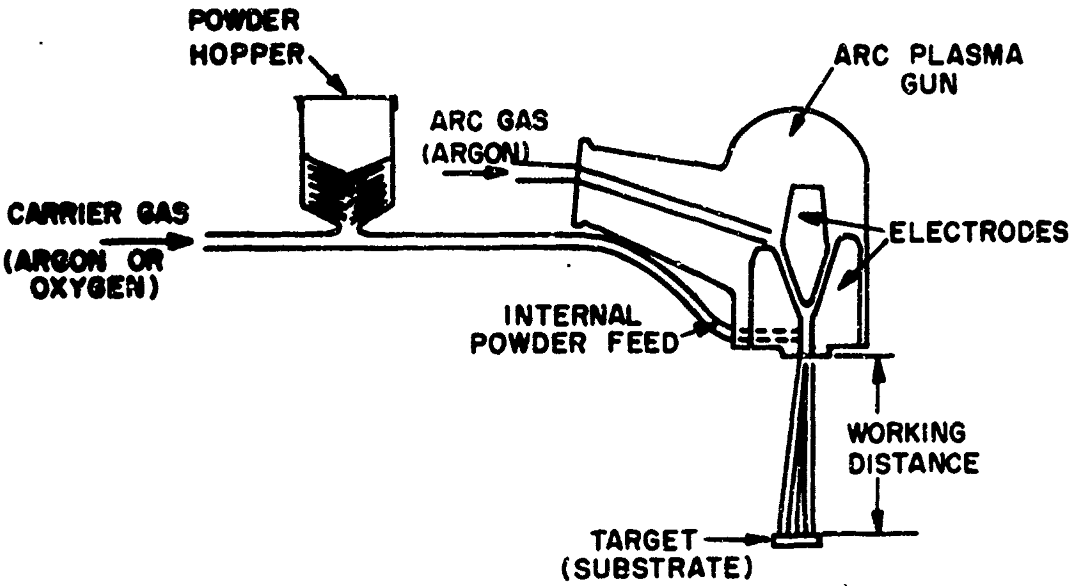


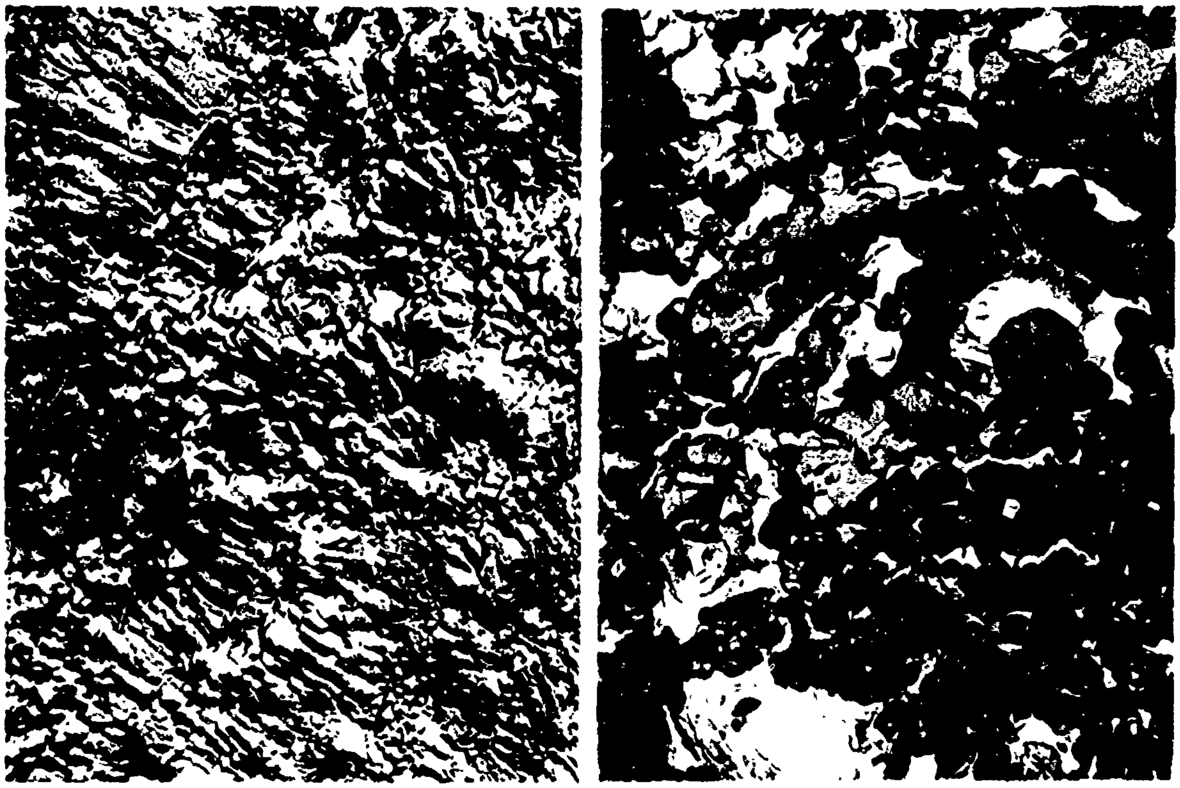

FIG. I ARC PLASMA DEPOSITION TECHNIQUE.

### Table XIII
### MAGNETIC PROPERTIES OF PAIRS OF TOROIDS CUT FROM SAME SUBSTRATE, BEFORE AND AFTER ANNEAL.

| | | Before Anneal | | | After Anneal | | |
|---|---|---|---|---|---|---|---|
| Powder | Toroid # | TC (ppm/°C) | $\mu'$ | $H_c$ (Oe) | $\mu'$ | $H_c$ (Oe) | Squareness Ratio |
| Fluid Bed | 1 | 0 | 34 | -- | 155 | 2.4 | .90 |
| | 2 | 0 | 55 | -- | | | |
| Flame Spray | 1 | -180 | 10 | -- | -- | -- | |
| | 2 | -180 | 16 | -- | 113 | 5.0 | .80 |
| Flame Spray | 1 | +40 | 25 | 21 | 138 | 3.1 | .84 |
| | 2 | +90 | 27 | 21 | 144 | 3.1 | .86 |
| Flame Spray | 1 | 0 | 11 | -- | 103 | 5.0 | .75 |
| | 2 | +70 | 11 | -- | 103 | 5.0 | .78 |
| Flame Spray | 1 | +80 | 11 | -- | 88 | 4.6 | .84 |
| | 2 | -- | 11 | -- | 91 | 5.0 | .83 |
| Flame Spray | 1 | -- | 24 | 20 | 95 | 3.2 | .65 |
| | 2 | -- | 23 | 24 | 103 | 3.2 | .67 |

### Table XIV
### REPRODUCIBILITY OF MAGNETIC PROPERTIES BY ARC PLASMA DEPOSITION.

| | | Before Anneal | | | After Anneal | | |
|---|---|---|---|---|---|---|---|
| Powder | Deposit # | TC (ppm/°C) | $\mu'$ | $H_c$ (Oe) | $\mu'$ | $H_c$ (Oe) | Squareness Ratio |
| Conv Spray Dried | 1 | +620 | 53 | 1.6 | 180 | .8 | .83 |
| | 2 | -- | 130 | 3.0 | 300 | 1.5 | .73 |
| Conv Spray Dried | 1 | -- | 42 | 12.0 | 160 | 3.0 | .80 |
| | 2 | -- | 53 | 11.0 | 220 | 2.3 | .78 |
| Flame Spray | 1 | +290 | 25 | -- | 62 | 5.0 | .85 |
| | 2 | +90 | 23 | -- | 97 | 3.5 | .85 |
| | 3 | +80 | 11 | -- | 88 | 4.6 | .84 |
| | 4 | -- | 11 | -- | 91 | 5.0 | .83 |
| Flame Spray | 1 | +140 | 25 | 22 | 120 | 3.5 | .89 |
| | 2 | +40 | 25 | 21 | 138 | 3.1 | .84 |
| | 3 | +90 | 27 | 21 | 140 | 3.1 | .86 |
| Flame Spray | 1 | +450 | 45 | 13 | 110 | 2.5 | .84 |
| | 2 | +400 | 38 | 15 | 120 | 3.1 | .80 |



(A) ARC PLASMA DEPOSITED FLUID BED FERRITE     5 $\mu$     (B) CONVENTIONALLY SINTERED FLUID BED FERRITE

FIG. 2 ELECTRON MICROSCOPE PHOTOGRAPH (5000 X) SHOWING THE EFFECTS OF ARC PLASMA DEPOSITION ON PARTICLE SHAPE.

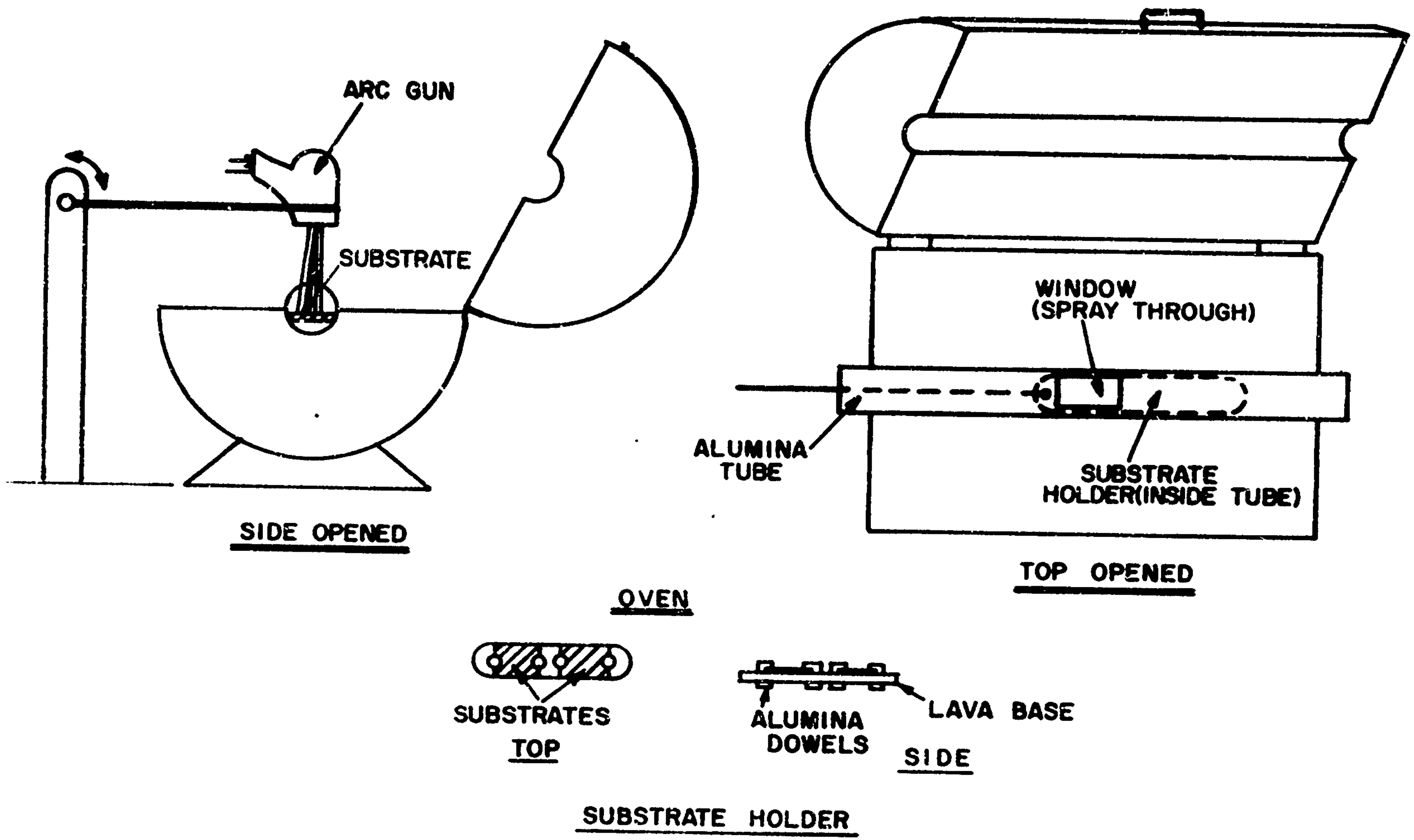
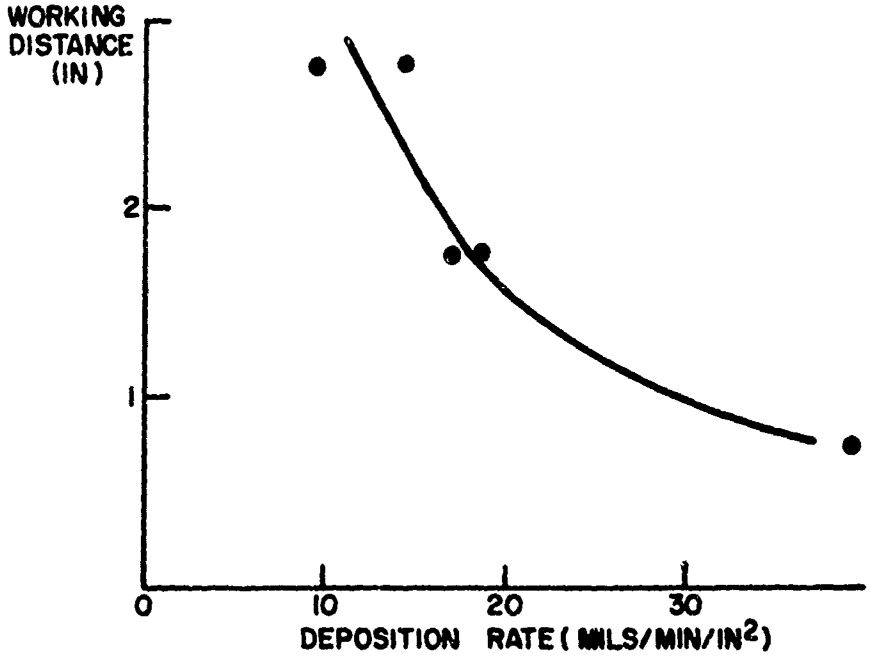
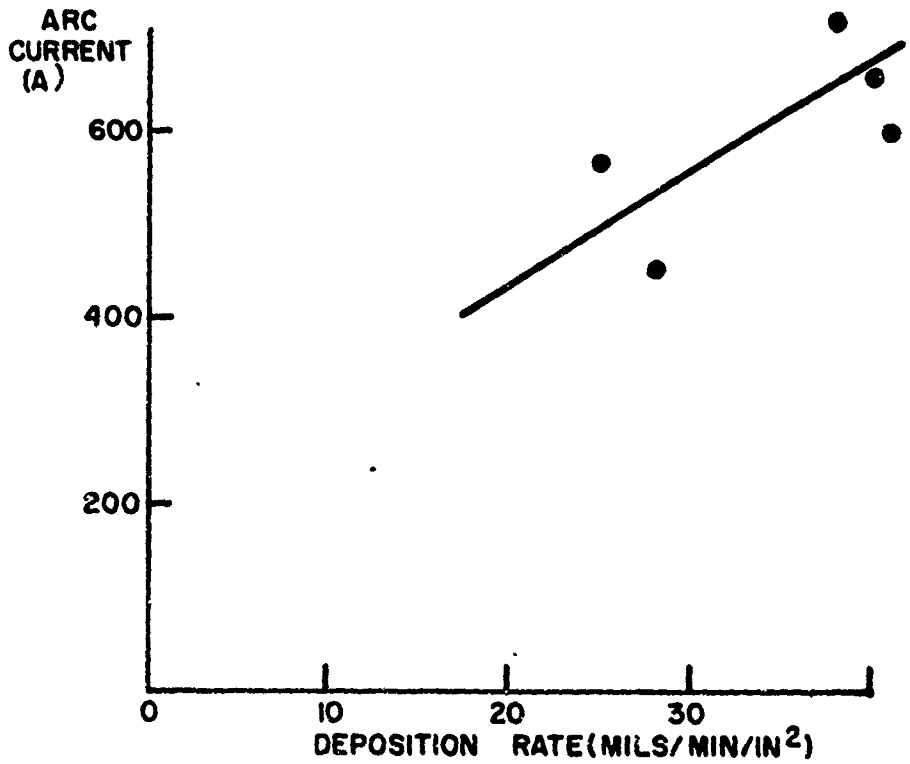



FIG. 3 OVEN USED TO HEAT SUBSTRATES PRIOR TO AND DURING ARC PLASMA DEPOSITION.




(a) DEPOSITION RATE AS A FUNCTION OF WORKING DISTANCE WITH AN ARC CURRENT OF 400 A.




(b) DEPOSITION RATE AS A FUNCTION OF ARC CURRENT, 1 1/2 IN WORKING DISTANCE.

FIG. 4 DEPOSITION RATE AS A FUNCTION OF WORKING DISTANCE AND ARC CURRENT.

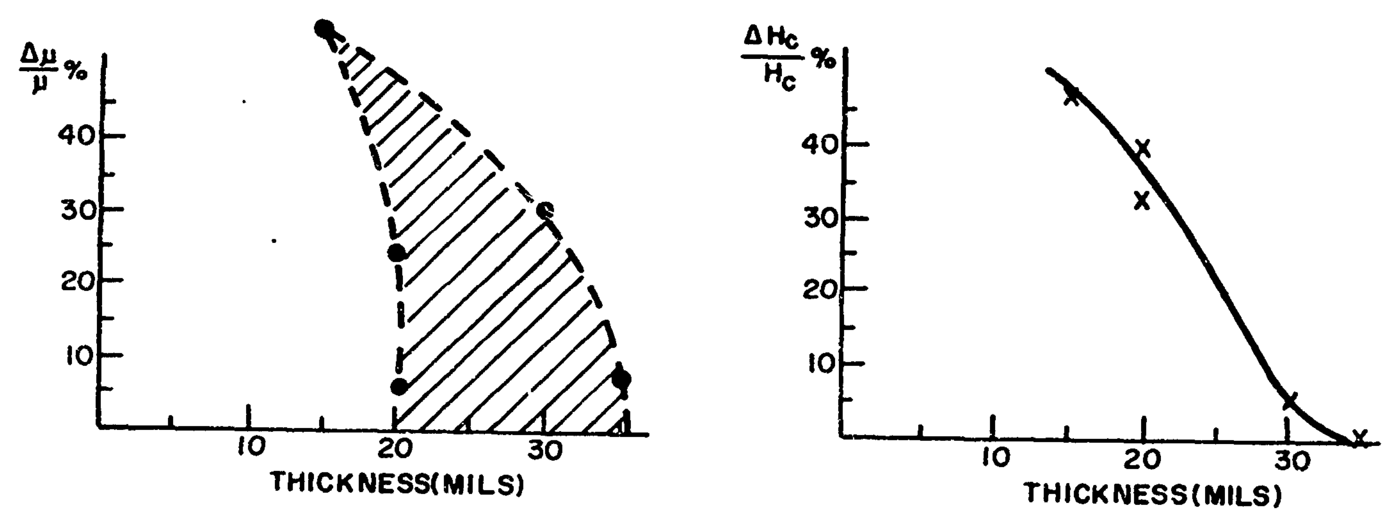



FIG.5 THE PERCENT VARIATION IN $\mu'$ AND $H_c$ AS A FUNCTION OF FERRITE FILM THICKNESS ON THE FERRITE-SUBSTRATE COMPOSITE AFTER ANNEAL.

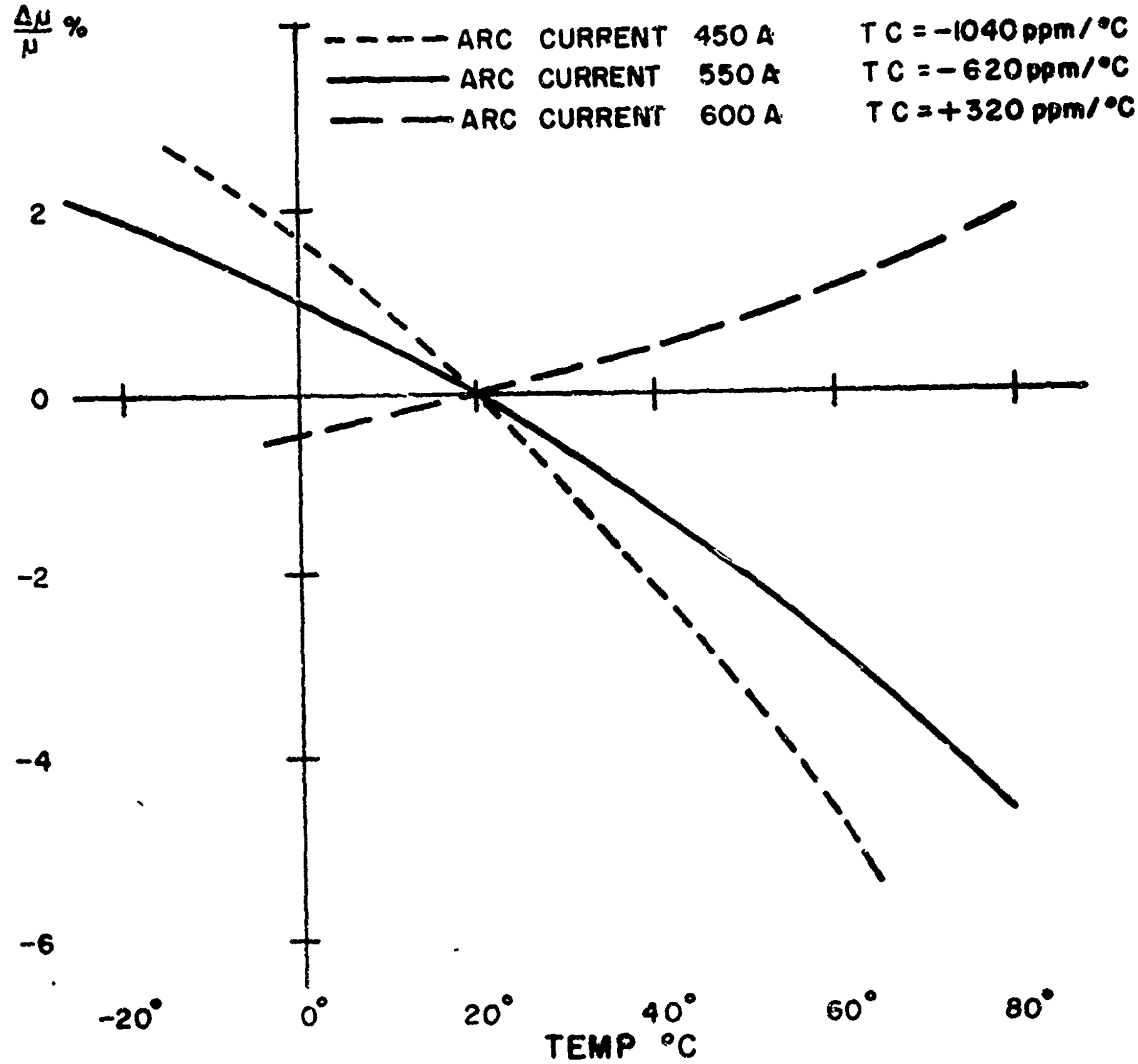



FIG. 6 EFFECT OF ARC CURRENT ON TC AT A WORKING DISTANCE OF 1-1/2 IN (CONVENTIONAL POWDER).

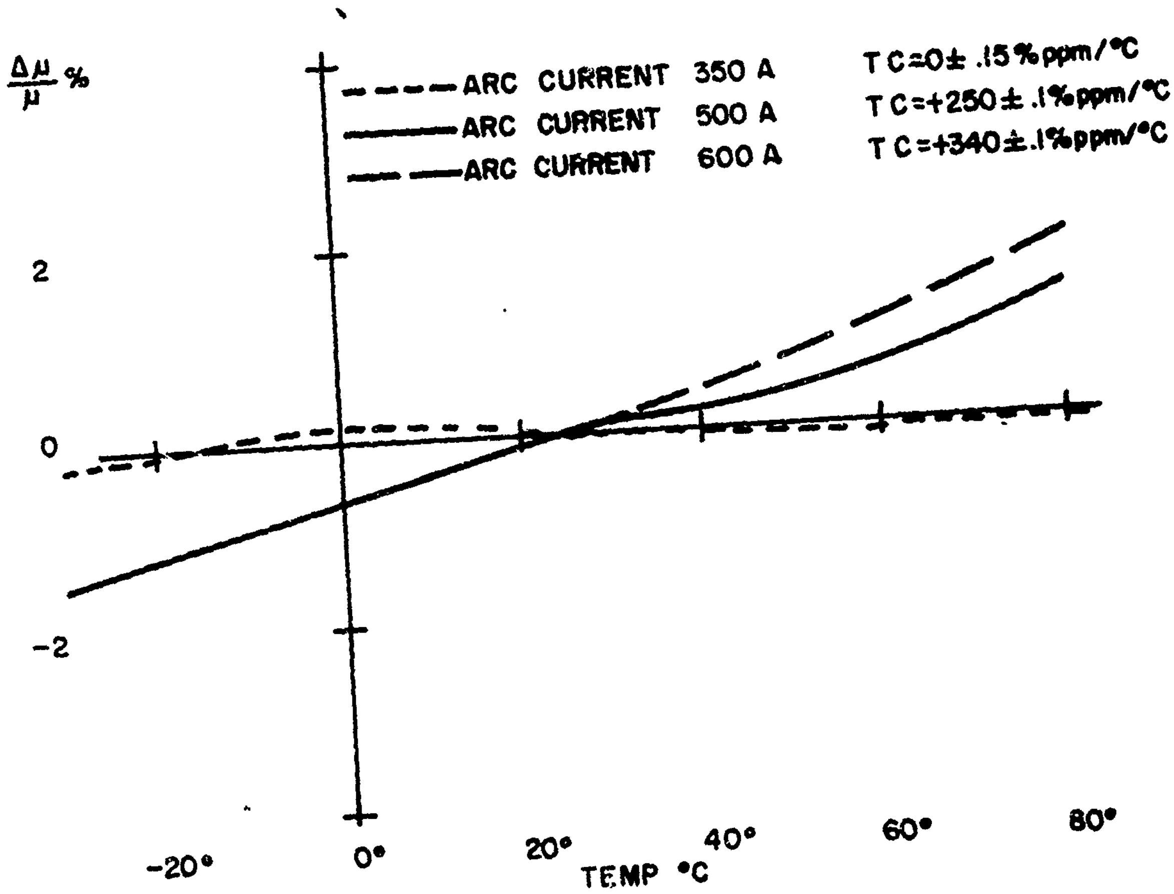



FIG. 7 EFFECTS OF ARC CURRENT ON TC AT A WORKING DISTANCE OF 1-1/2 IN (FLUID BED POWDER).